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A RAPID CEMENT CONTROL TEST FOR CEMENT TREATED BASES

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During the past few years the necessity has arisen for a rapid field test which can be used to determine cement distribution in treated bases during the course of highway construction. The advent of higher quality aggregates and the lowering of cement content, as well as the increased use of road mixing methods, made it imperative that such a test be developed.

The stabilization of soils and base materials with Portland cement essentially started in California in 1937. In these early beginnings mixing was largely accomplished by plows, disks and harrow methods. During this period fairly high cement contents were employed analogous to Eastern "soil cement" practice. While this mixing method often did not provide a uniform mixture of cement and soil, the relatively high cement content usually assured at least some degree of hardening throughout the material.

In the early 1940's improvements in cement treatment were instituted which included the use of modified bituminous mix plants of the pugmill type. At that time specifications were also adopted requiring the use of processed aggregates having grading limits similar to those for untreated base material. Minimum

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seven day compressive strength requirements were established and the product was named cement treated base or CTB. The general improvement in aggregate quality and the setting of strength specifications for the treated mixture had the effect of lowering cement contents considerably from those previously used. With this reduction in cement, the problem of obtaining better uniformity in mixing assumed greater importance. The use of the central mixing plant where the ingredients are proportioned by weight and mixed intimately in a "pug", generally provided a needed improvement in cement distribution.

The late 40's and early 50's saw the introduction of specifications which permitted road mixing methods, employing machines designed for the purpose, as a substitute for central plant methods. Most contractors abandoned the plant methods largely because of the possibilities for increased production, particularly where multiple road mixing units could be employed. In addition, road mixing also released plant facilities for concurrent production of bituminous mixes.

From the engineer's standpoint, however, the lack of a rapid testing method in the road mixing type of operation made it more difficult and often next to impossible to control several important variables in the construction of cement treated bases. Such things as the uniformity of material in the windrow, uniformity of cement spread in advance of mixing, uniformity and thoroughness of mixing due to high forward speeds of the mixer, size of the windrow in relation to the capacity of the mixer, depth of mixing as well as the mechanical condition of the mixer itself can all profoundly influence the uniformity of cement distribution.

Recent experiences have indicated that while plant operations are not subject to as many variables as road mixing, serious variations in cement distribution of the final mixture sometimes result from malfunctioning or poorly equipped plants. Difficulties from the latter are particularly evident with the continuous mix type plants when rigorous and reliable flow controls and ingredient feed interlocks are not provided.

Unfortunately the nonuniformity introduced by the variables affecting the various types of mixing operations cannot be controlled by normal methods of bulk quantity measurements. Actually only systematic quantitative tests performed on random samples can provide the basis for evaluation of the efficiency of cement treated base operations.

The California Division of Highways laboratory has in the past investigated several test methods which are aimed at determining the amount of cement in a sample of the mix. These tests are:

(1) Chemical analysis in accordance with ASTM designation D-806;

(2) Compressive strength tests to determine uniformity; and

(3) Electrical conductivity measurements.

For various reasons these methods did not prove satisfactory for monitoring field mixing operations. The chemical analysis method must be performed in a well equipped laboratory by a trained chemist and the compressive strength method requires at least a week to obtain results. These are distinct disadvantages which preclude effective use for field control. The electrical conductivity method, which is a broad modification of a test developed for portland cement concrete by Mr. L. R. Chada of India, proved to be a vast improvement over the other two methods in that it can be performed on the project and requires only about three hours to obtain results. However, the electrical method was found to be sensitive to soluble salt inclusions in the aggregates and this often precluded the use of the test in California coastal regions where the aggregates had been exposed to ocean water or in certain inland areas where so-called "alkali" soils abound.

In the summer of 1957 our attention was directed to a method employed in England* for evaluating cement distribution by means of acid digestion and titration. The method as originally developed was used for soil-cement mixtures and required considerable modification before it could be applied with any degree of accuracy to the cement treated base mixtures used in California. However, with modifications, a satisfactory test has been developed known as the "Acid-Base Titration Method", which may be easily and rapidly performed at the construction site.

The test is arranged, with respect to time, so that up to eight individual samples may be concurrently tested by one operator. Test specimens weighing 300 grams are prepared from the freshly

*The Determination of the Cement Content of Soil-Cement III. An Investigation of Some of the Factors Involved by P. T. Sherwood. Journal of Applied Chemistry, 1957, 7, Nov. 1957.

mixed CTB and placed in plastic containers (approximately 2 qt. size). 200 milliliters of 3 normal hydrochloric acid (HCl) are then introduced into each sample and a standardized stirring procedure is followed for 18 minutes after which the acid is diluted to one normal concentration. In this process the acid neutralizes the cement and in the process there is a reduction in the acidity or Ph of the total solution. This reduction in acidity is measured by back titration with sodium hydroxide (NaOH) and the amount of NaOH required is inversely proportional to the cement content of the test specimen.

At 30 minutes after the first introduction of HCl, a 100 milliliter aliquot portion of the residual acid is transferred to a beaker and prepared for titration by adding a small quantity of phenolphthalein solution. Titration is accomplished with 1 normal sodium hydroxide solution using a burette, graduated to 0.2 ml. The alkali is added slowly to the aliquot portion until a permanent red color is attained by the solution. The reading on the burette in milliliters will then indicate the amount of NaOH that was needed to neutralize the residual acid.

In order to convert this burette reading into terms of percent of cement it is first necessary to establish a relationship for the particular cement, aggregates and water used on the project. This is accomplished by developing a "standard curve" from tests on prepared samples of the aggregate containing known amounts of the particular cement being used. Normally the curve is a straight line; therefore, it is usually only necessary to perform tests on duplicate aggregate blank specimens (containing 0 percent of cement) and on duplicate specimens containing 5 percent cement. For convenience the titration values obtained are plotted on a graph against cement content as illustrated in Figure 3. Since a straight line relationship exists between milliliters of NaOH and percent cement, it is merely necessary to draw a line between the averages of the paired tests. This then provides the standard curve from which the percentages of cement in individual field samples taken from the project may be determined.

Field and laboratory trials with the Acid-Base Titration method involving approximately 1,700 tests and 22 construction projects, indicates that the test provides a very satisfactory means of monitoring the distribution of cement. The test is comparatively simple and results can be obtained on groups of eight samples in about 45 minutes, excluding sampling and preparation time. Statistical studies show that the test is reliable and indicates a standard error of estimate of only ± 0.2 percent (cement).

Unfortunately this procedure cannot be used where the CTB aggregates contain significant amounts of such substances as limestone, calcite and dolomite. These minerals react to hydrochloric acid in a manner similar to portland cement and therefore cause false cement determinations. This condition is readily discernible and limiting test values are established which indicates to the operator when the test should not be used.

As a result of this difficulty a supplemental test procedure, referred to as the "Constant Neutralization Method" was developed by the Materials and Research Department for use on projects where aggregates susceptible to acid exist. While this method utilizes the same apparatus as the acid-base test, it is somewhat more time consuming and tedious and therefore less desirable in cases where aggregates reacting to the acid are not involved. It does however, provide a satisfactory means of control testing in the cases where the acid-base test cannot be used.

The constant neutralization method can be performed on a maximum number of four 300-gram test specimens at one time. The specimens are placed in a plastic container to which 250 milliliters of water and a small quantity of phenolphthalein solution are added. The solution will immediately turn pink due to the release of hydration products from the cement. Then using a burette containing 3 normal hydrochloric acid the operator continuously adds acid and stirs the mixture to maintain a colorless solution for a timed interval of one hour. He must not add any more acid than is just necessary to remove the pink color, for any excess acid will attack the susceptible aggregates. Likewise, if he does not add enough acid the hydration products going into solution from the cement will be arrested thereby affecting the end results. Conscientious attention to these details, by the operator, for the full time period of one hour is absolutely necessary in this test. The amount of hydrochloric acid used in this process is determined from volume measurements with a burette or by weight. Experience has shown that the amount is directly proportional to the cement content of the treated base sample. The cement content of the field sample is determined from a standard curve developed for the project using the constant neutralization method as illustrated in Figure 4.

The sampling and the preparation of representative specimens of cement treated bases presents a special problem in connection with both test procedures. In California, cement treated base aggregates are usually composed of coarse granular, graded materials. With such material the greater portion of the cement normally combines with the fines leaving a lesser amount adhering to the coarser particles. This makes it essential that tests be performed on specimens having the same ratio of coarse to fines as exists in the road or windrow after mixing. Testing the fine portion alone would not solve this difficulty as the amount of cement adhering to the coarse particles is somewhat variable depending on moisture content. It would also be necessary to prepare multiple standard curves representing various gradings or varying test specimen weights in order to accommodate changes in the coarse to fine ratio. It has been found simpler and more accurate to include, in the test specimen, the coarse aggregate up to the 1-1/2" maximum size.

For the preparation of reliable test specimens, the field samples must be at least 3 kilograms in weight in order to be reasonably representative of the bulk material. Each sample is immediately separated on the 3/8" sieve* and the proportions of the retained to passing calculated (including any retained 1-1/2" material). Any aggregate retained on the 1-1/2" sieve is then wasted and the two sizes of material (± 3/8") are recombined to form a 300-gram test specimen having typical proportions of plus

*Consideration was originally given to separating the sample on the No. 4 sieve. However, the larger size is easier and faster for field operations where moist samples must be hand sieved.

to minus 3/8". In the latter process, equivalent amounts by weight of the plus 3/8" aggregate are substituted for any wasted rock larger than 1-1/2" in order to maintain the correct relationship of coarse to fines characteristic of the original field sample.

Both of the titration procedures are incorporated into one standard test method (Calif. No. 338) for the California Materials Manual and are now being applied in field operations. The 1960 California Standard Specifications permit a maximum variation from the planned cement content of ± 0.6% cement for road mixing and ± 0.4% cement for plant mixing operations.

The equipment for performing the method has been conveniently arranged in kit form as shown in Figure 5. A chest has been designed for containing the numerous individual items (i.e. burettes, flasks, plastic containers, beakers, etc.) to minimize breakage and loss. Plastic containers have been used wherever possible including the 5 gallon carboys for holding acid and base working solutions. The entire equipment is easily and safely transportable in a station wagon type vehicle for field testing operations.

Since this method involves the use of acids and reagents it should be performed by technicians trained in laboratory techniques. While corrosive liquids are involved and present some hazard to personnel if improperly used, the Division of Highways Safety Engineer has approved the method on the condition that use be limited to trained laboratory personnel. It has therefore, been arranged that engineers from the District Materials Department will perform the test as needed on various projects within their respective districts.

On numerous occasions in field applications, the new test has demonstrated a particular usefulness as a tool in isolating causes of poor distribution occurring during CTB operations. Through the application of various sampling techniques and some ingenuity on the part of the engineer, it is often possible to "pinpoint" and correct sources of difficulty.

The choice of two sampling schemes on a road mixing operation, for example, can be used to differentiate between the uniformity of cement application to a windrow in advance of mixing and the effectiveness with which the machine integrates the cement-aggregate-water mixture. The distribution of cement between samples taken transversely across the windrow or spread out material is primarily a function of the machine's mixing ability. On the other hand samples taken of the freshly mixed material longitudinally at various stations down the path of mixing relate to the evenness of cement deposition.

It has been our experience that contractors are willing to build good roads but are wary of spending money unnecessarily. However, when a contractor is shown direct evidence that he is not getting a satisfactory product, and particularly where tangible indications can be given regarding the source of deficiency, he is much more willing to make alterations in this operation. Figures 6, 7 and 8 illustrate three actual cases where corrective measures were taken with dramatic results, on the basis of our analysis of test data.

Figure 6 displays an example involving a batch type plant mix operation. In this case titration tests performed on samples obtained from the pugmill at approximately 3 minute intervals, representing individual batches, indicated considerable variation in cement content with a number of the test results falling outside of the allowable limits ($\pm$ 0.4%) from the planned cement content of 3.5% (see curve labeled "before"). Incidentally bulk quantity measurements spanning over the period when the samples were taken, indicated an overall cement content that was acceptable with respect to the planned value, which further demonstrates the ineffectiveness of this method from a quality control standpoint. When nonuniformity is encountered during the sampling of successive batches, such as on this project, it is usually indicative of difficulties being experienced in the batching of materials. As a consequence, after careful examination of both the aggregate and cement batching operations, it was found that the cement weighing scale was not only out of calibration but was "over capacity" for this job to the point where it was nearly impossible for the plant operator to weigh with any degree of accuracy. When this was pointed out to the contractor, he immediately purchased a new scale, had it calibrated and installed on the plant. Subsequent retests indicated a vast improvement in cement distribution as shown by the curve labeled "after" in Figure 6.

The example illustrated in Figure 7 concerns a continuous mix type plant operation in which samples, taken at approximately 30 second intervals from the output belt, indicated fluctuations in cement content from a high of about 5% to a low of 2.5%

(see curve labeled 4" auger). With a planned cement content of 4.0% for the project, this range was outside of tolerances in both directions. Again it appeared that the materials feed was at fault and since the aggregate was moving uniformly to the pugmill, the cement feed was given close scrutiny. This particular plant utilized a 4 inch diameter, 4 inch pitch calibrated screw conveyor from the cement silo to the pugmill. From indications at the silo, the cement was frequently bridging over the auger causing alternate lapses and surges in the feed. After the installation and calibration of a larger 9 inch diameter, 4 inch pitch auger this problem was solved as evidenced by the subsequent titration test results shown in the figure.

In a particular road mixing operation, given as the final example in Figure 8, the cement distribution was extremely poor. While the planned cement content was 3.0%, test results on samples taken transversely across the "spread out" material indicated a high of 6% and a low 1.5% (see curve for road mixer X). Since the results were based upon transverse samples, the nonuniformity apparently resulted from the machine itself or the manner in which it was operated. The contractor was permitted to try several corrective measures. However, it soon became apparent to the resident engineer that all was to no avail. The resident engineer then instructed the contractor that he could no longer use that particular machine on the project. When another machine was subsequently used, the cement distribution became satisfactory as indicated by the curve for machine Y.

It is our belief that continued use of the titration test on a statewide basis will bring inestimable benefits to the State by helping to secure cement treated bases of consistently uniform quality. With uniformity will come savings in cement as accurate control will result in equal quality with less cement.

Acknowledgments

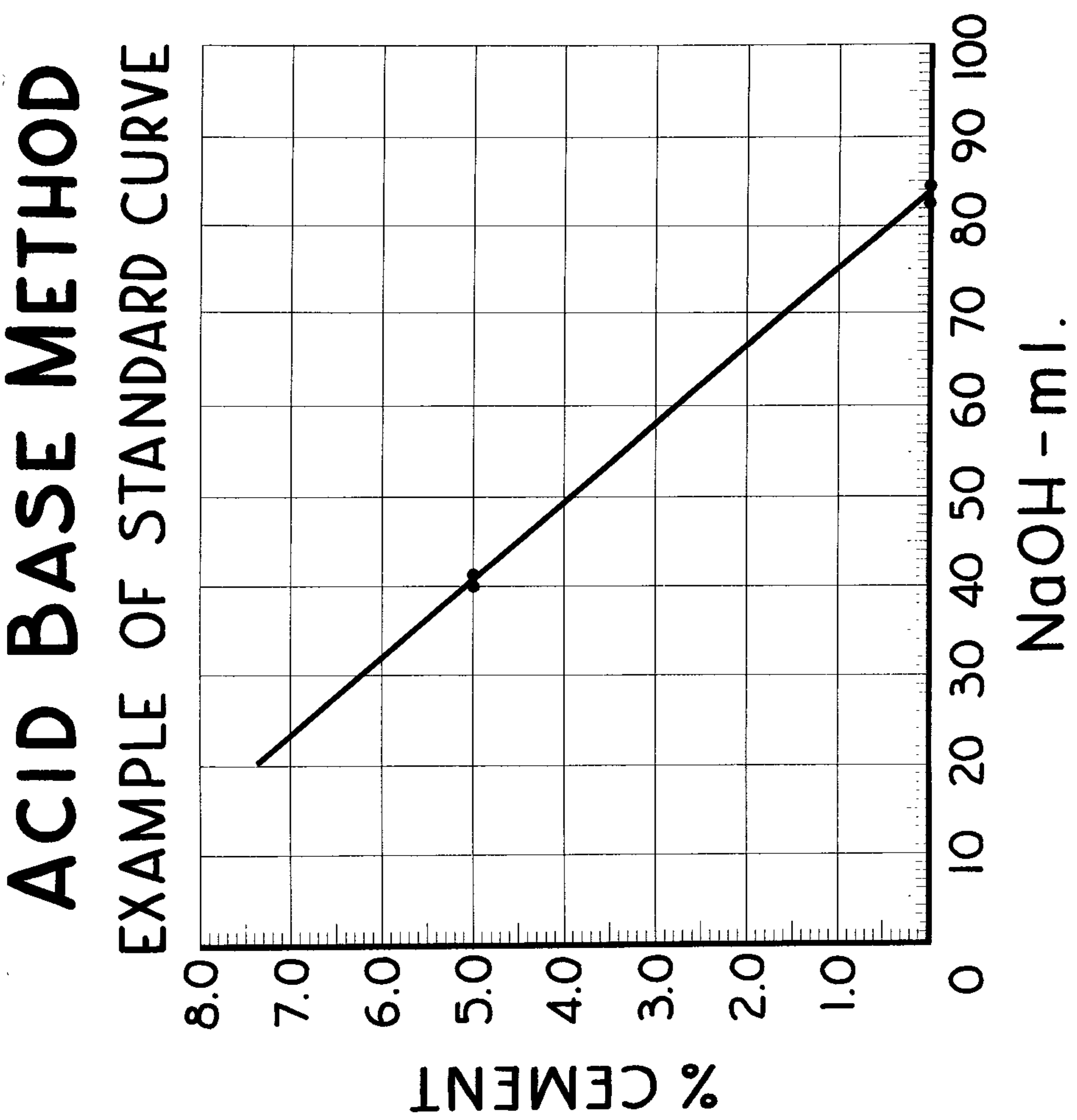
This article covering the development of a titration method for determining the cement distribution in cement treated bases represents the combined efforts of a number of people. The work was carried out in the Materials and Research Department under the general direction of Mr. Ernest Zube, Supervising Materials and Research Engineer, and Mr. Clyde G. Gates, Senior Materials and Research Engineer.

Special acknowledgment is made to Mr. John Borchert who originated the idea for the constant neutralization test.

FIGURE 1 — The acid-base titration test can also be easily performed at the construction site.

FIGURE 2 — Layout for the performance of the constant neutralization procedure is illustrated above.

FIGURE 3


ACID BASE METHOD
EXAMPLE OF STANDARD CURVE
% CEMENT
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
10
20
30
40
50
60
70
80
90
100
NaOH - ml.

CONSTANT NEUTRALIZATION METHOD

EXAMPLE OF STANDARD CURVE

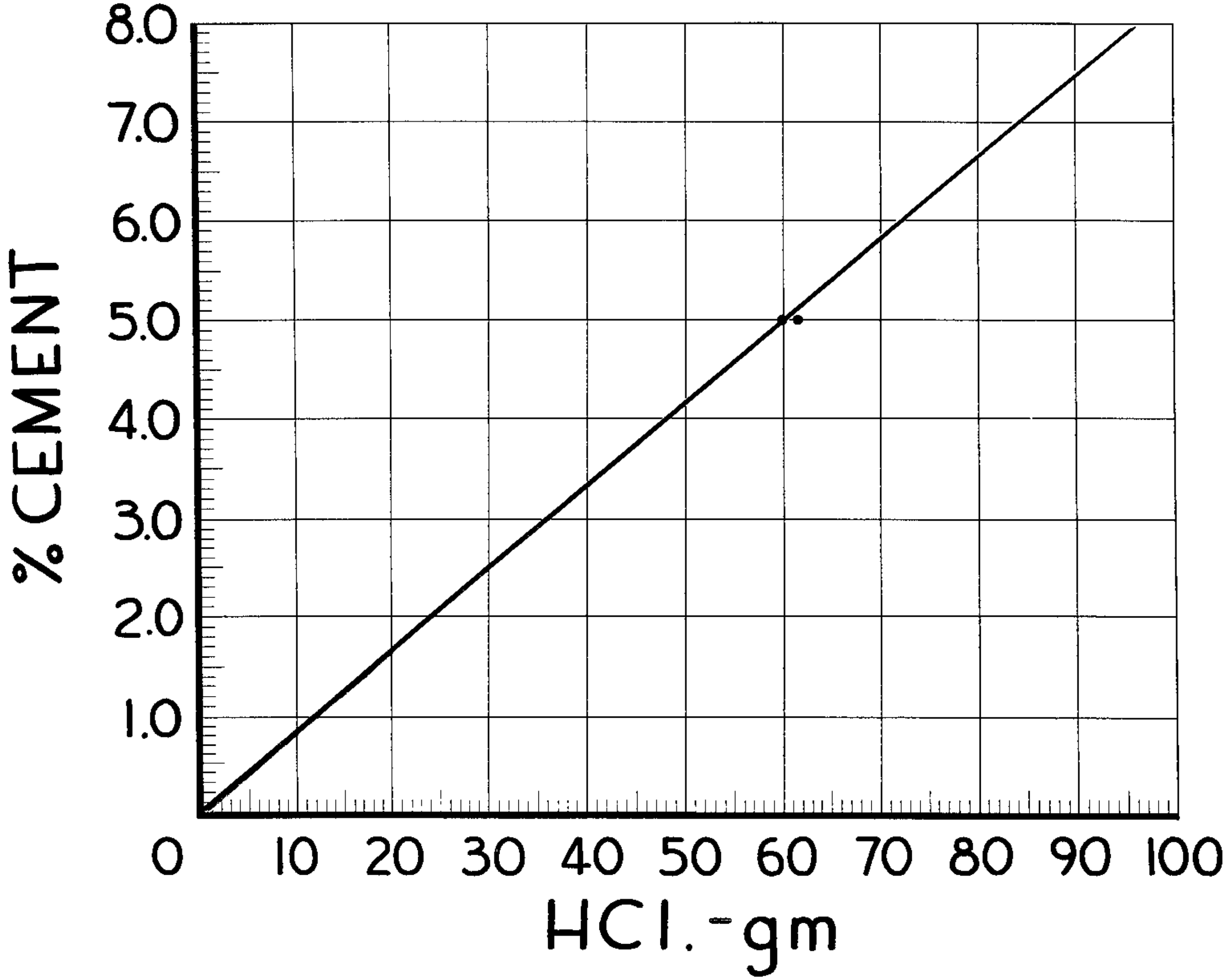
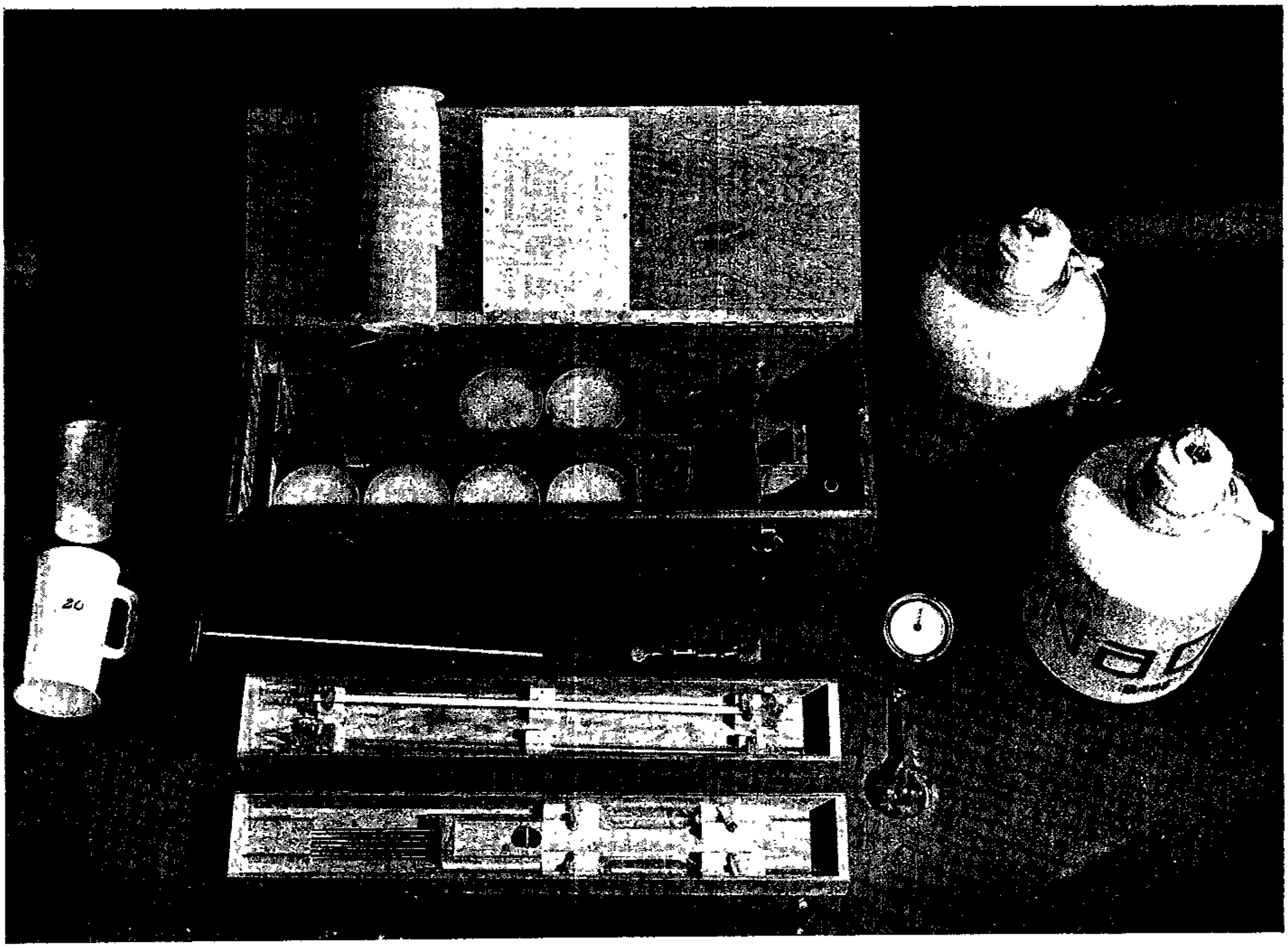


FIGURE 5



The field kit for the titration test.

FIGURE 6

PROJECT A
BATCH PLANT CTB OPERATION

Example of improved distribution in CTB mixture by replacement of inaccurate cement scale.

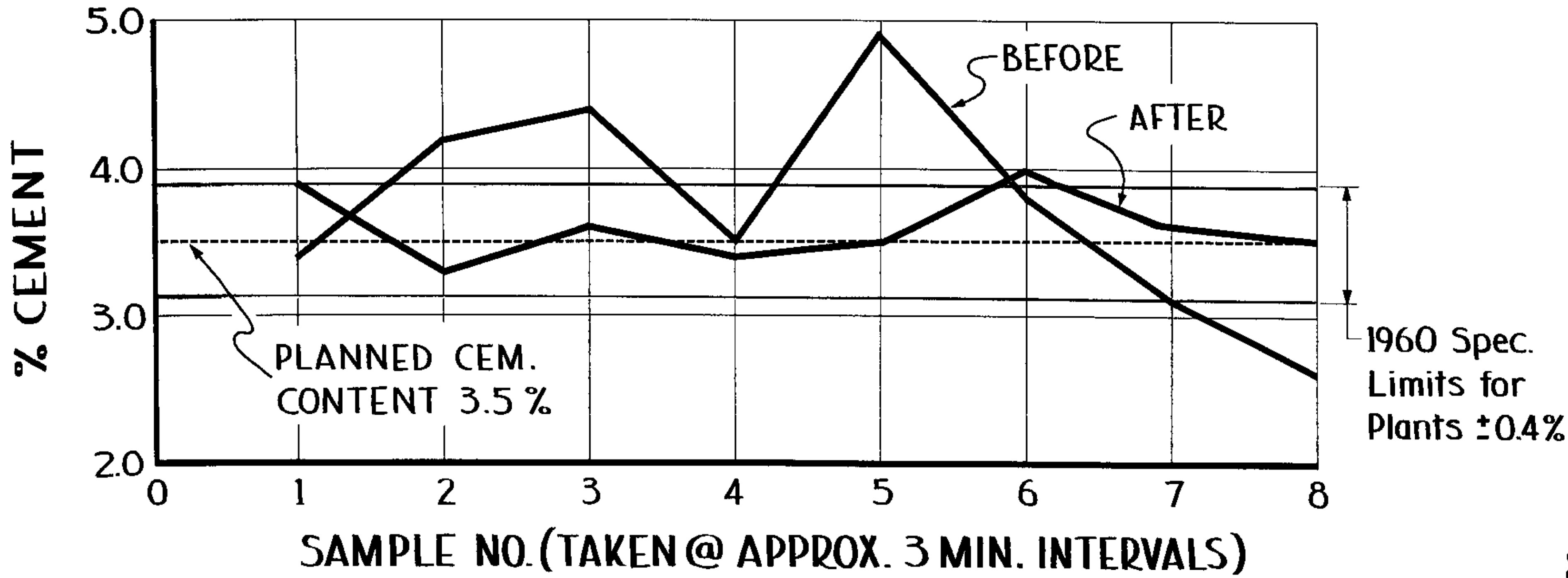
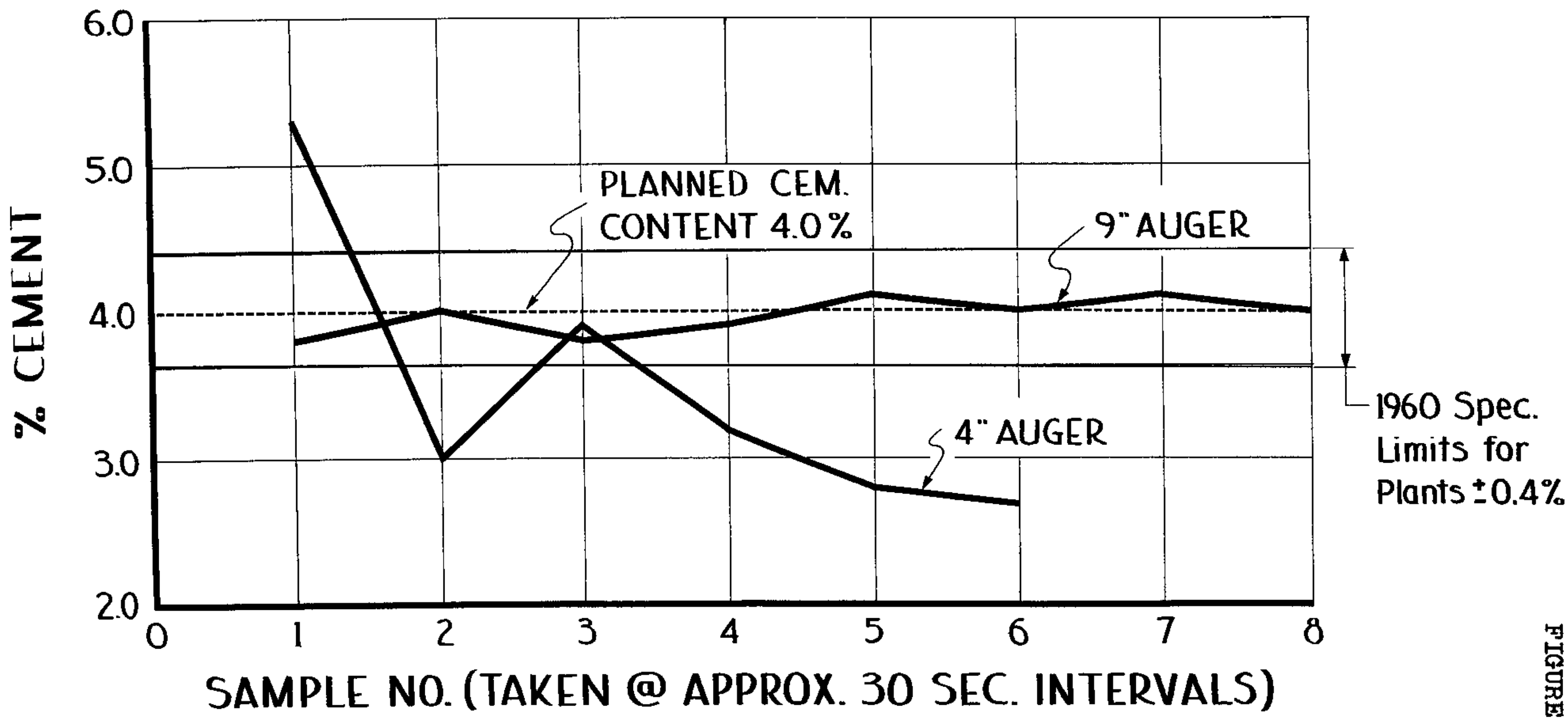


FIGURE 7

PROJECT B

CONTINUOUS MIX CTB PLANT

Example of improved distribution by changing cement feed auger from 4" dia., 4" pitch to 9" dia., 4" pitch.



PROJECT C

ROADMIXING CTB OPERATION

Example of improved distribution by the replacement of a poorly functioning machine with one which operates properly.

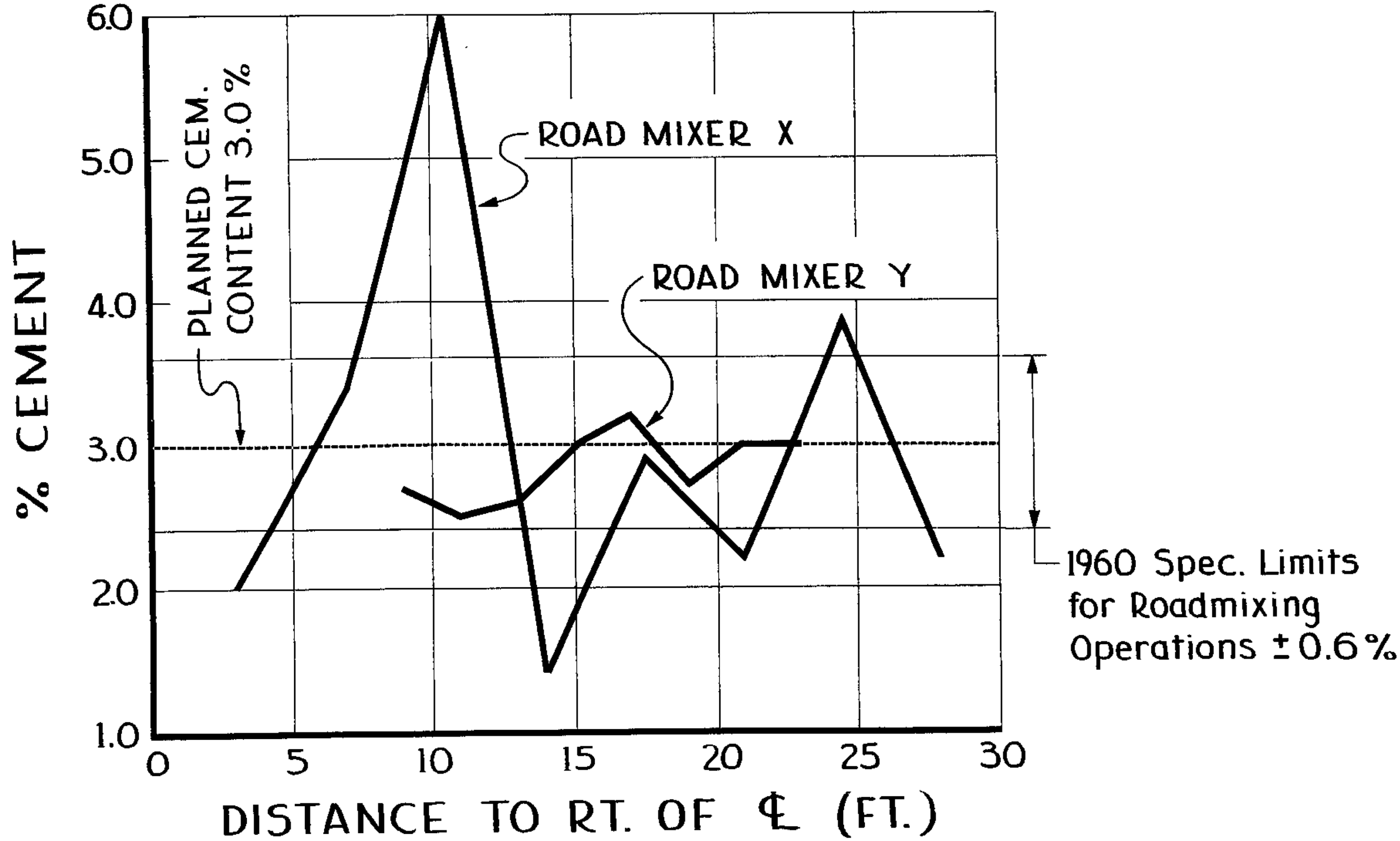


FIGURE 8